Sandia Microsensors for Industrial Process Monitoring

Alex Robinson, Sandia National Laboratories

Acknowledgements, Sandia:

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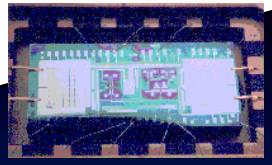
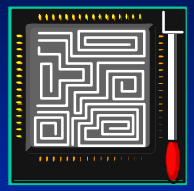
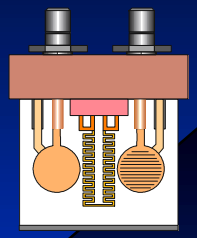
Miniature Sensors, Processors, and Communication Systems

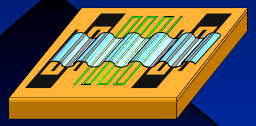


Micromachines, Microelectronics

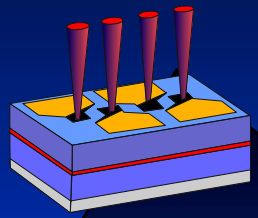


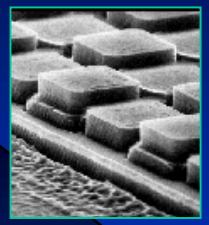
Microsensors





Photonics, Microwave Circuits





Microelectronics Development Laboratory
MDL

Over 30,000 ft² of clean room, 0.6 μm CMOS Fabrication Facility

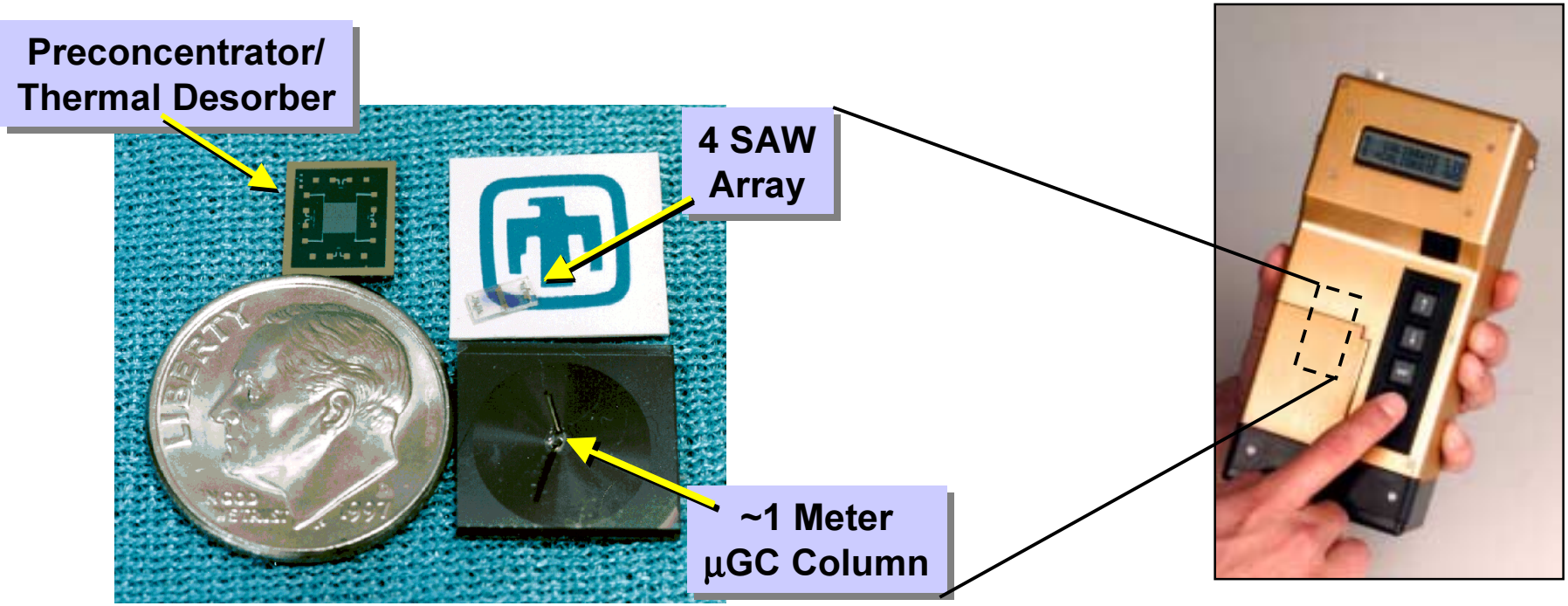
Integrated Materials Research Laboratory
IMRL

Materials Fabrication and Characterization, including Plasma Deposition and Surface and Interface State Characterization

Compound Semiconductor Research Laboratory
CSRL

MOCVD, MBE Deposition, Electron Beam Lithography, Reactive Ion Beam Etching

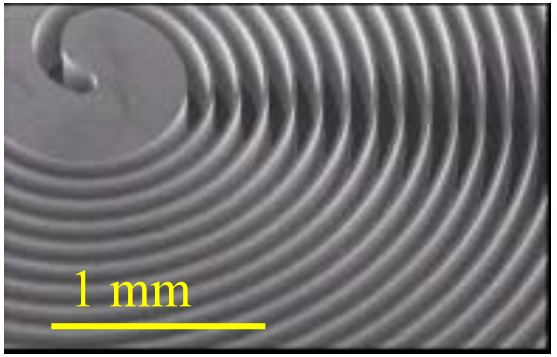
Microfabricated Components for Sandia's µChemLab



Silicon Microfabricated Chromatography Columns

Wall-Coated μ-Column

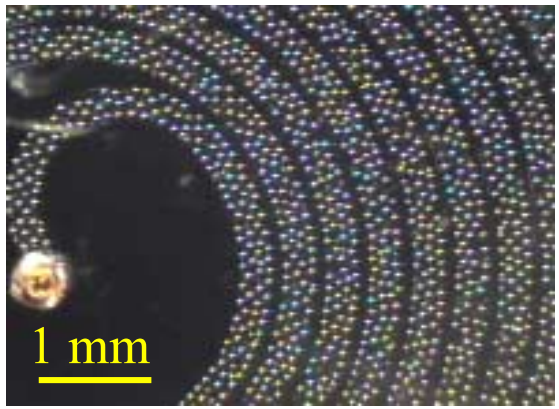
- Variety of polymer functionalities can be covalently bonded to walls
- High thermal diffusion coefficient and small mass allow rapid temperature ramping
- Retention tailored for VOCs to heavy molecules (> b.p. 440°C)



channel dimensions:
400 μm x 100 μm x 86 cm

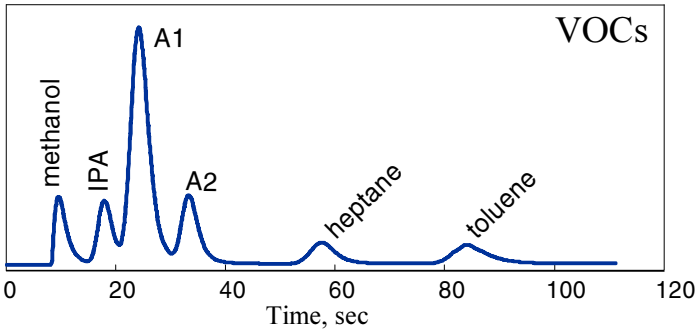
Packable μ-Column

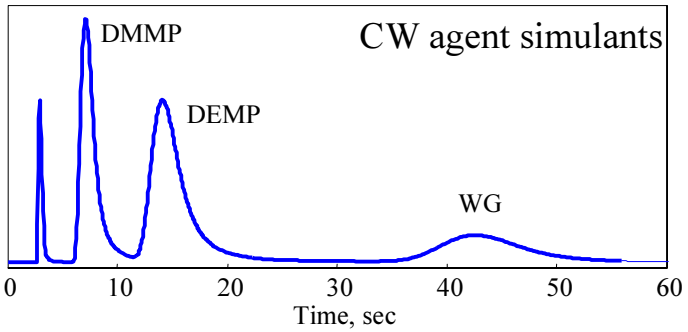
- Enables use of existing column packing materials and technology
- Provides necessary retention for separation of gases and light VOCs
- Patent application filed

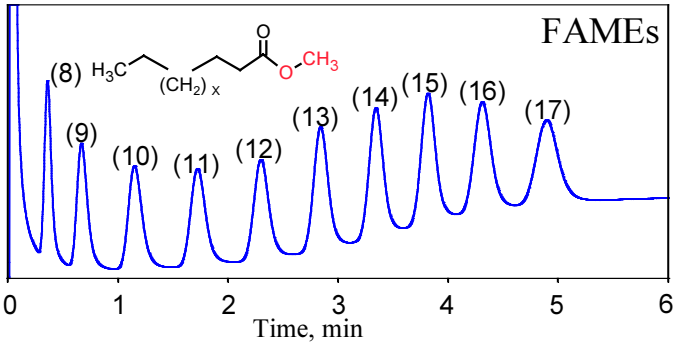


channel dimensions:
300 μm x 300 μm x 29 cm

Broad Applicability







μ-Columns Tailored to Specific Applications

- Light gases: CO, CO_2, C_1-C_4
- Pharmaceutical drying, VOCs
- Chemical warfare agents/simulants
- Biological analysis, FAMEs

Specificity Achieved Through

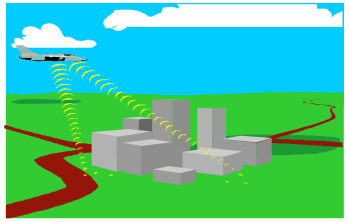
- Specific wall coatings or packings
- Column length
- Rapid temperature cycling

µChemLab Application Strategy

Sensitive
Selective
Fast

Low Power
Hand Held
Low Cost
Versatile

Non-proliferation



Counter Terrorism

Military (CW/BW)

Biomedical Diagnostics

Industrial Processes

Environmental

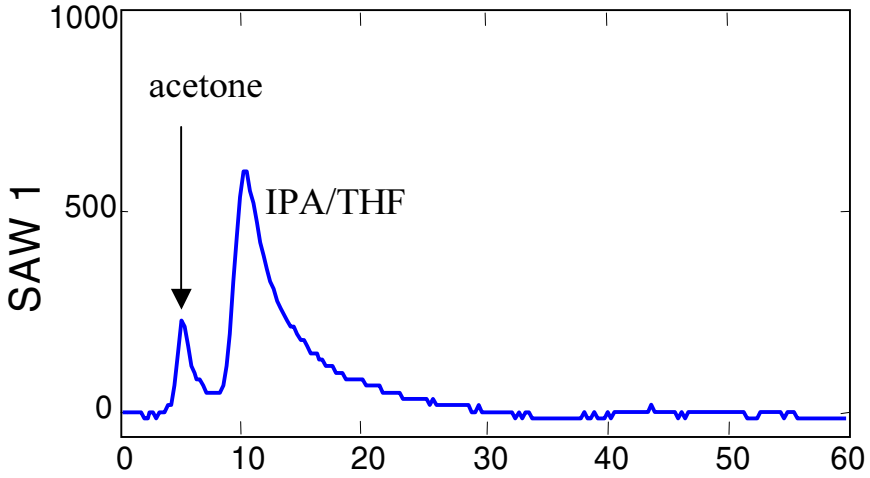
Industrial Hygiene

Food and Water Safety

Pharmaceutical Industry

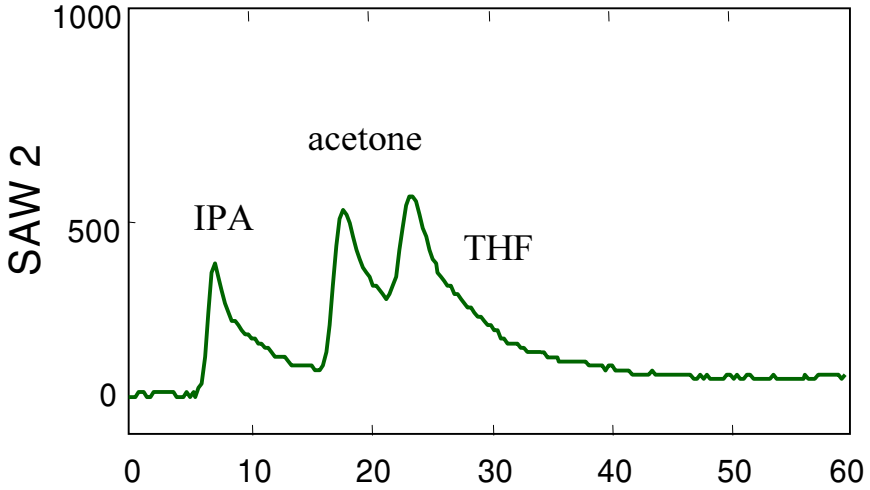
- Delivered to Bristol-Myers Squibb June 2002
- Air carrier, portable, on-line or sample bag analysis
- Pharmaceutical applications in fermentation, synthesis, and product drying
- Correlate peak height/area to solvent concentrations. End process within 2 minutes of end point.
- On-going collaboration

Pharmaceutical Industry



Packed μ-Column
ramped from 35 to 60 °C over 60 sec

Test Mixture
2000 ppm isopropanol
3000 ppm acetone
1000 ppm THF



TEC Cooled μ-Column
ramped from 5.5 to 30 °C over 60 sec

Petrochemical Industry

ARU (Acetylene Refinery Unit)

Problems

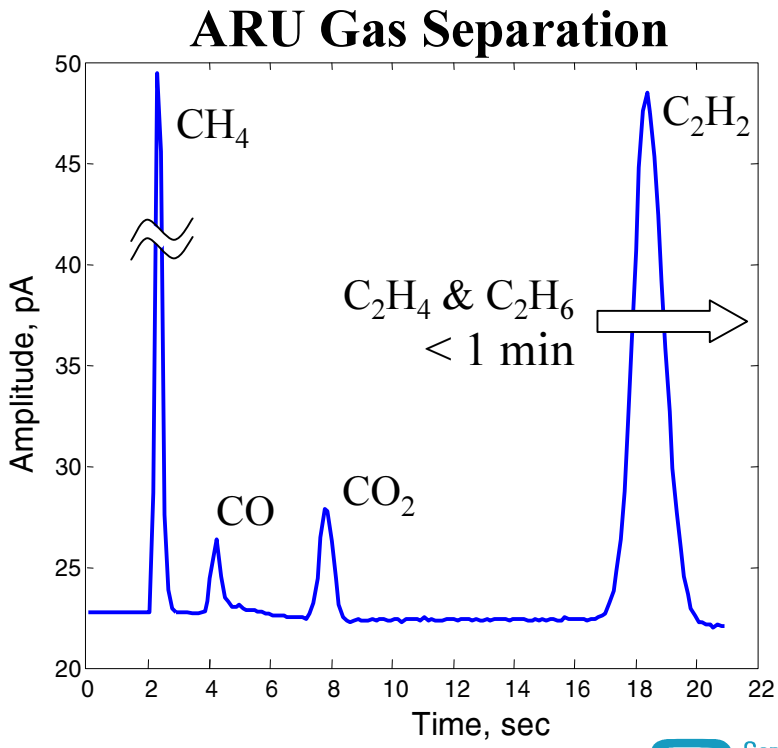
- Reactor runaway requires dumping to flare ($40K/hour)
- Current process methods are expensive to implement and maintain

Goals

- Reduce product loss
- Increase yield efficiency
- Reduce overall energy consumption
- Lower instrumentation costs

29 cm packed μ-column

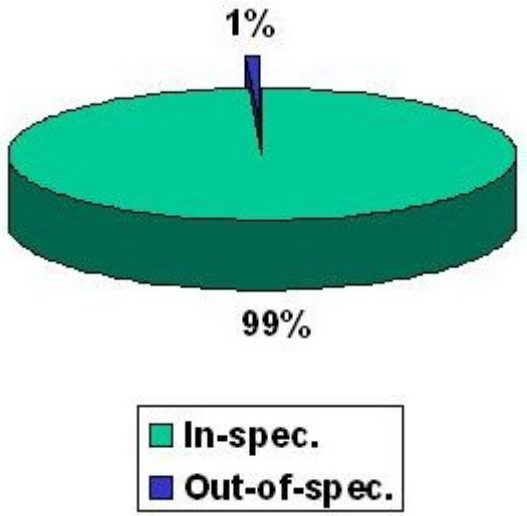
He discharge μ-detector



Petrochemical Industry

- Faster analysis cycle-time can reduce product loss from 12.6 M lbs. to 2.5 M lbs. in single ARU
- Equivalent to $400K/year/ARU in reduced product loss and improved energy use
- ~$16.6 M/year worldwide
- Similar processes could use same technology base

Present Ethylene Product Yield *



*Data from a Phillips ARU over a 16-mo. interval

Fossil Energy

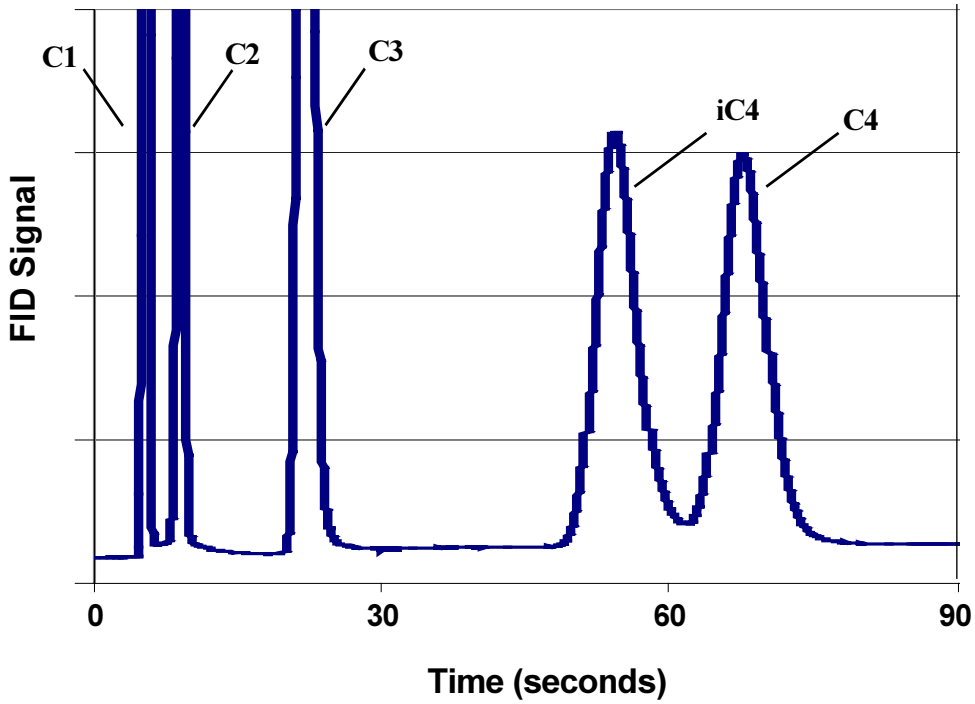
Goal

- Lower price point for natural gas analyzers to increase sensor distribution ($5K)

Benefits

- Lower up-front, operating, and maintenance costs
- 10% improvement in unaccounted for BTU will benefit gas transfer industry by $45M/year
- Improved use through near real-time knowledge of physical gas properties (composition, dew point, heating value)

Separation of Natural Gas Standard



19 cm micro-column packed with porous polymer beads.

Fossil Energy

Goal

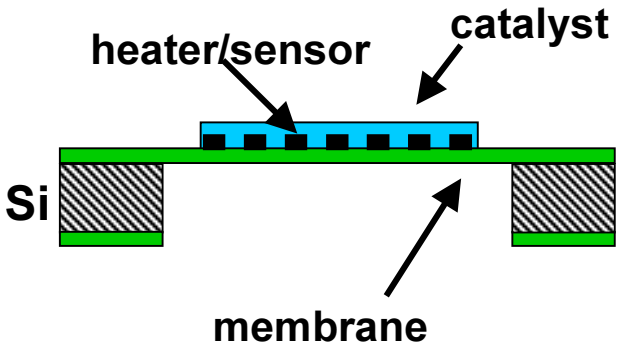
- Real-time measurement of fuel composition and lower heating value (LHV)

Approach

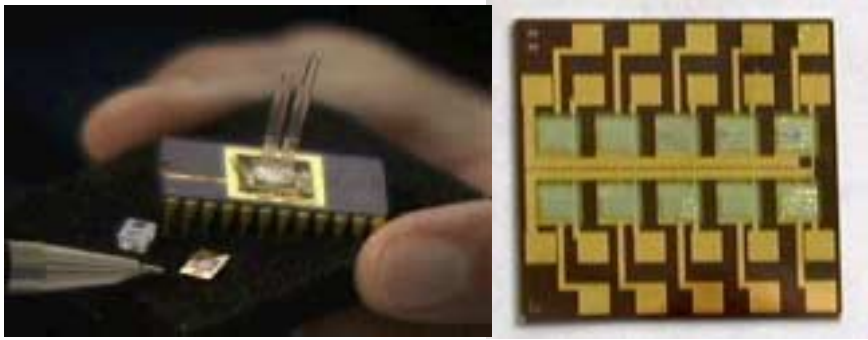
- Current: Pattern recognition on an array of microhotplate calorimeters
- Next: microcalorimeter array; microGC and microFID

Benefits

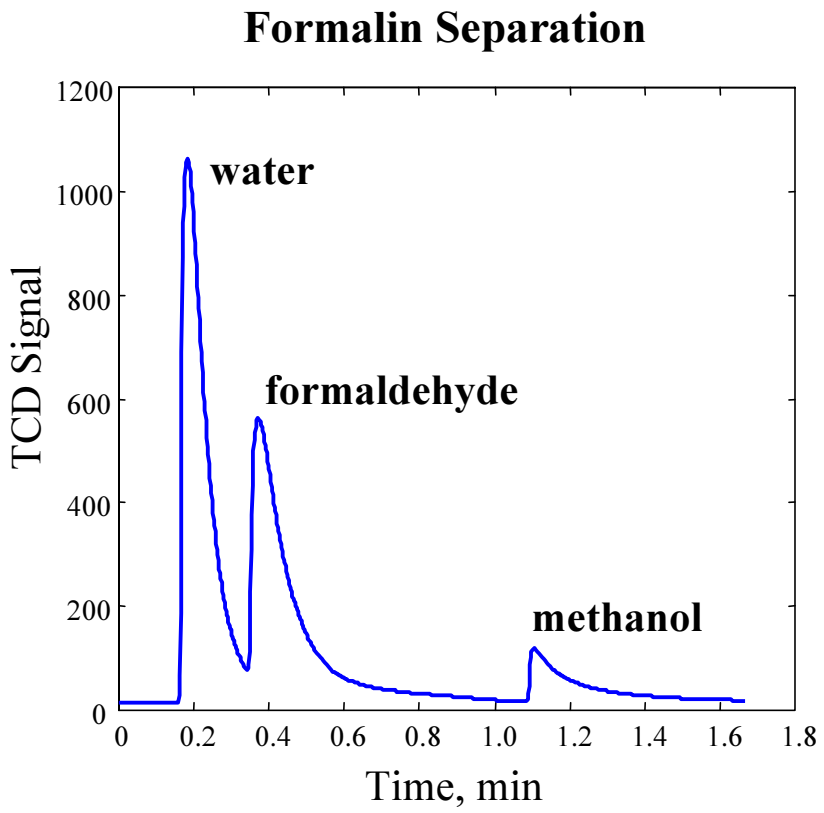
- Intelligent combustion in turbines, increasing efficiency and lowering emissions



A minimum of two hotplates for LHV



Forest Industry



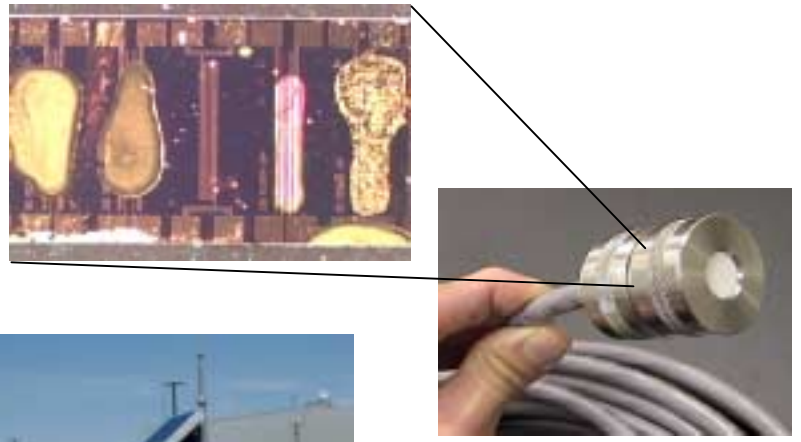
0.2 uL liquid injection, TCD detector
29 cm packed micro-column, Carboxen 1000
Carrier: He, 3.8 mL min, 30 psi
Oven: 120 degrees C

Formaldehyde Monitoring in Pulp and Paper Manufacturing

- EPA requires monitoring of stack emissions
- OSHA requires monitoring of work floor concentrations
- Higher processing temperature increases production rates and reduces costs, but raises emission levels

Water Monitoring

- Chemiresistor "sniffer" provides real-time in-situ detection of volatile contaminants in air, soil, and water
- Reduce costs associated with long-term environmental stewardship
- Cleanup and Fines = $$
- Sensors deployed and tested at Nevada Test Site, Edwards Air Force Base, CA, and Chemical Waste Landfill, NM



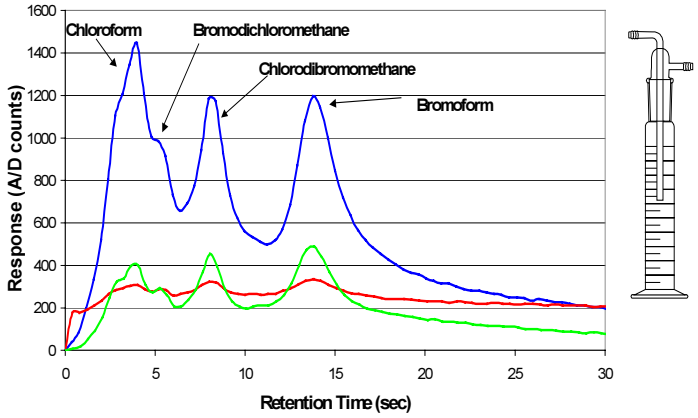
In-Situ Chemiresistor Sensor

Edwards AFB, CA

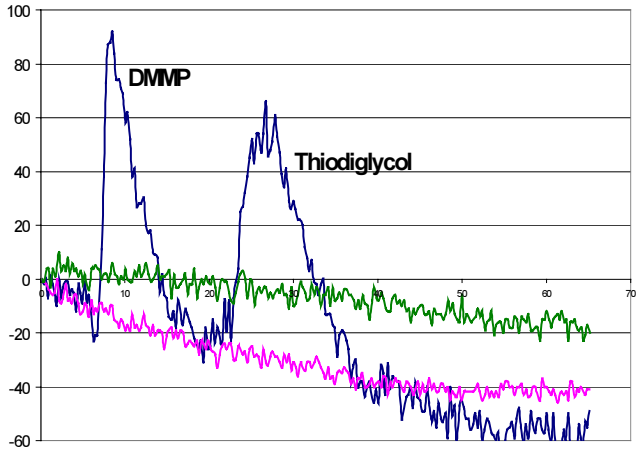
Chemical Waste Landfill, NM

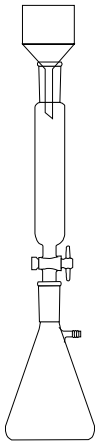
Water Monitoring

Air Stripping
(*Volatiles*)



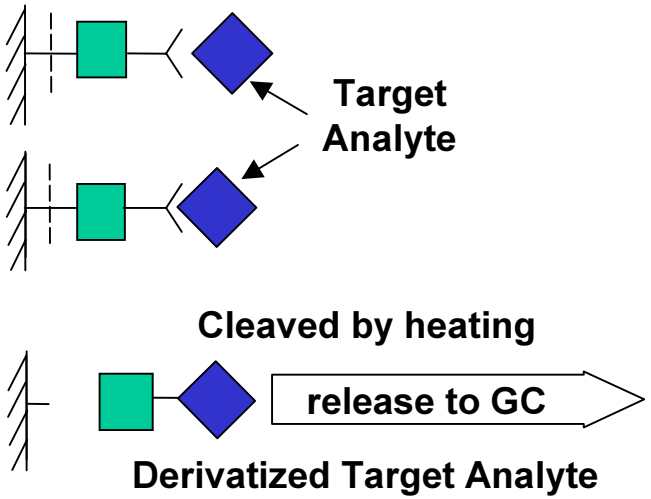
Solid Phase Extraction
(*Semivolatiles*)





Condensed Phase Collection Methods

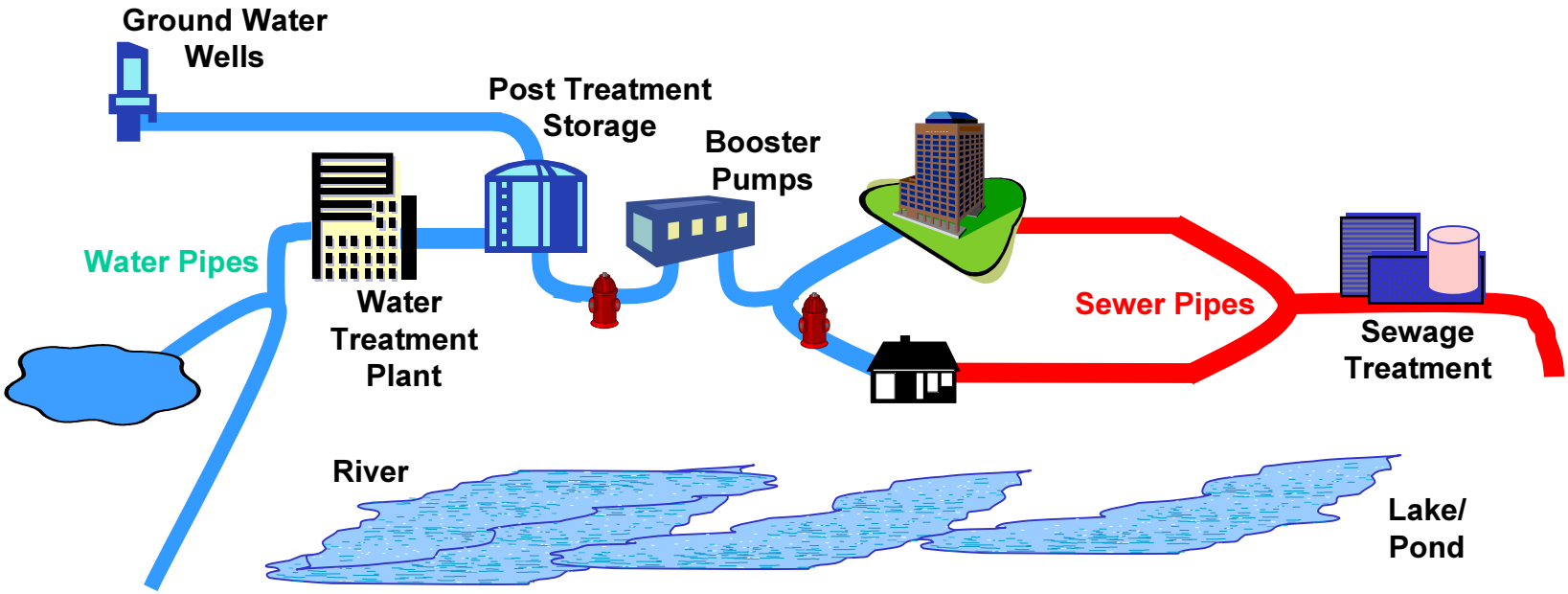
Trap Tag and Release
(*biofouling*)



Water Monitoring

Purified Municipal Water has been the primary focus. Many of these technologies will have cross-cutting opportunities or applications within:

surface supply water, ground water, waste water, food, steam supply systems, process water, and pharmaceuticals



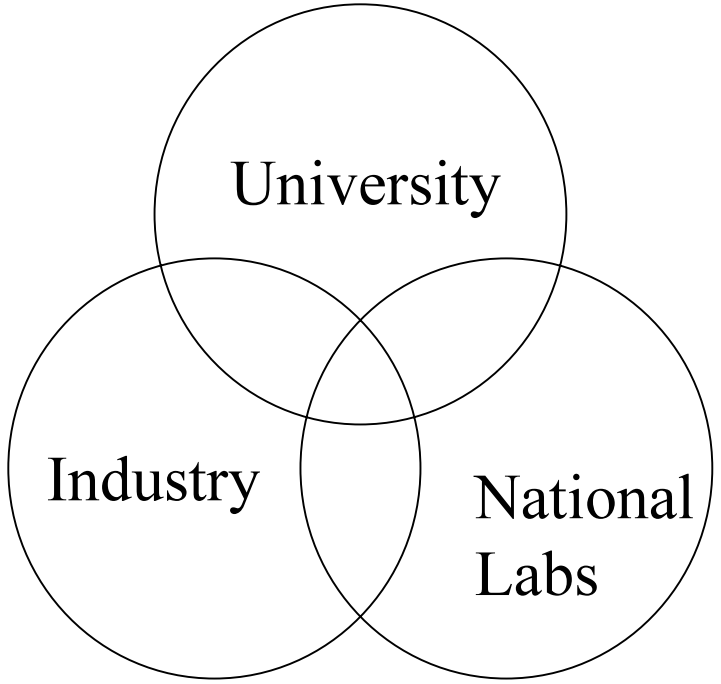
Expand Industry/National Labs Collaboration

Issue

- National Labs' research slow to reach industry
- National Labs' research often complimentary to University research
- Bridging gap from lab to vendor to industry

One solution

- DOE sponsorship of National Labs to University research centers



Expand Industry/National Labs Collaboration

- *Example*: CPAC-Center for Process Analytical Chemistry
  University of Washington, Seattle

- CPAC has an established track record of fostering interactions academic/industrial/national laboratory, aiming to bridge the gap between basic research and full-scale process/product development.

- In direct response to industry needs and priorities CPAC initiated a program to facilitate technology transfer in all phases of development through to commercialization

- ***The 2003 CPAC Summer Institute, July 16-18, 2003***
  "Reducing the time from discovery to commercialization"

Expand Industry/National Labs Collaboration

Proposal

- DOE provide multi-user sponsorship to CPAC for 5+ National Labs

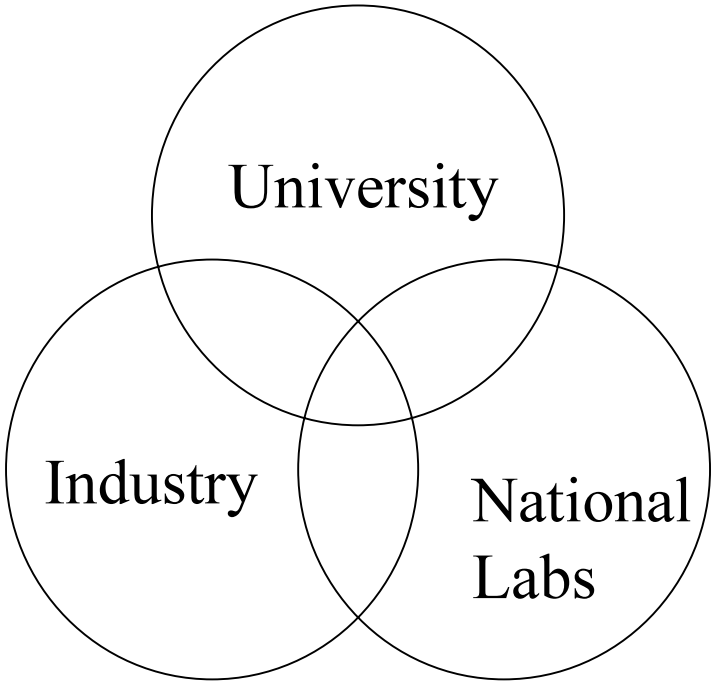
(Sandia, Los Alamos, Oak Ridge, Savannah River, Pacific Northwest*)*

Cost

- $200K/year total membership fee
- $40K/year per lab for travel and personnel time
- Total cost: $400K/year

Benefit

- More National Lab involvement in organization geared towards technology development and transfer to commercial vendors and industry
- More research with stronger leveraging
- Energy and other cost savings to industry



* current CPAC members

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